STUDY OF THE PESTICIDE RESIDUES IN RICE MATRIX BY LIQUID CHROMATOGRAPHY COUPLED WITH MASS SPECTROMETER

SHORT SYNOPSIS

Submitted in partial fulfillment of the requirement of the degree of

DOCTOR OF PHILOSOPHY

by

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Date: 30/01/2025

DECLARATION BY THE SCHOLAR

I hereby declare that the short synopsis entitled "Study of the pesticide residues in rice matrix by liquid chromatography coupled with mass spectrometer" being submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in the Department of Sciences under the School of Sciences of Manav Rachna University, Faridabad during the academic year 2024-2025.

It is the work that is expected to be carried out under the supervision of (Dr.) Meena Kapahi, Professor and co-supervision of (Dr.) Roopa Rani, Assistant Professor

Further, it is declared that the short synopsis is within the plagiarism limit and copy of the same is attached as per the University norms.

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CERTIFICATE BY SUPERVISOR(S)

This is to certify that, the short synopsis entitled "Study of the pesticide residues in rice matrix by liquid chromatography coupled with mass spectrometer" is an original piece of work done by research scholar Deepak Ashok Temak under the supervision of (Dr.) Meena Kapahi, Professor and co-supervision of (Dr.) Roopa Rani, Assistant Professor for the degree of Doctor of Philosophy in the Department of Sciences under the School of Sciences of Manav Rachna University, Faridabad during the academic year2024- 2025.

Further, it is certified that the short synopsis is within the plagiarism limit and copy of the same is attached as per the University norms.

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Abstract

To determine pesticide residue in rice matrices at low concentration, there is a need for suitable analytical method. In the current study, the dispersive solid-liquid extraction method has been employed to monitor different types of pesticides by liquid chromatography with tandem mass spectrometry (LC-MS/MS) electrospray ionization (ESI) technique with a minimum run time. The QuEChERS method for pesticide residues extraction and determination in cereals is used. More than 50 different pesticides are monitored for their compliance with it tolerance set by different regulatory bodies. The method validation protocol is utilized as per SANTE -11312/2021 guidelines by evaluating different parameters such as linearity, specificity, LOD, LOQ, recovery, repeatability, reproducibility, ion ratio and robustness.

Keywords: Pesticide Residue, Dispersive Solid-Liquid Extraction, LC-MS/MS, EU Guideline, SANTE 11312 guideline, Maximum Residual Limit, QuEChERS

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1. Introduction:

Cereal is the main food of every common man. Among all, rice is the most eat up cereal grain and a necessary food for more than 66 % of the world population. However, the major five states are West Bengal, Utter Pradesh, Punjab, Tamilnadu and Andhra Pradesh. West Bengal produce 14 % of the total rice produced in India [1-3].

Significant rice production is done by applying supplementary pesticides directly on soil and standing crops. Insect pest damage in terms of yield loss ranges from 25 to 30 % around the world due to the use of insecticides, herbicides and etc. in rice cultivation [4]. Not only insects but sometimes heavy rainfall, yellow stem borer, rice gall midge and weeds and disease infestation is responsible for decrease to rice production and loss. They also contribute to the loss in grain yield and quality of rice [5]. More than 914 different formulations are used for controlling pest's namely organophosphate, synthetic parathyroid neonicotinoids, triazole fungicides, and herbicides have been registered for the cultivation of rice [6].

1.1 Adverse effects of pesticides:

Due to the application of pesticide on agriculture crops to control different pest which is responsible for economic damage. After application pesticide they enter in to the ecosystem through runoff method, leaching, bio-degradation, bioaccumulation etc [7]. Subsequent are the studies of harmful impact of pesticides in environment:

1.1.1 Effect on soil system:

Application of pesticides residue in soil, poses negative effects like pollution, resulting into the destruction of the useful microorganisms, creating impact of reduction of soil quality and generate difficult situation for crop production [8].

1.1.2 Effect of aquatic ecosystem:

The significant fragment of environment is water and it is highly acknowledged that with lacking water there shall be no existence of life, but at the same time water gets polluted due the pesticide through discharge and superficial runoff method. Polluted water is very injurious to the water environment as well as human wellbeing [9].

The process of bio-magnification may result in the deposition or accumulation of pesticides content in different organs of animals [10-13].

1.1.3 Effect on vegetables and fruits:

Since the use of pesticides can be at all stages of crop production right from beginning like sowing, harvesting, storing and this leads to higher chance of

contamination of crops which ultimately are consumed by human or animals. This results in harmful impact on germination of seeds also and the final production too [14-16].

1.1.4 Effect on human being:

Crops which are pesticide contaminated when consumed by human being can pose serious health impacts. Even if the crop is harvested and washed, some of the pesticide residue may remain over crops or fruits, which are finally consumed by human. Pesticide can cause cancer, asthma, nervous system damage, reproductive disorder, etc. Therefore, there lies a necessity to examine the pesticide residue present over crops like rice, wheat, etc which has a major part of daily diet of human being. The permissible limit of pesticide residue generally lies among 0.01 ppm to 2.5 ppm [17 – 26].

2. Literature Review:

In current research work, the most common pesticide residues which is found in food matrix has been selected for developing a liquid chromatography coupled with tandem mass spectrometric (LC-MS/MS) method and method validation of the developed method in order to reliably estimate them in food matrix while adopting the latest regulatory and laboratory guidelines. The comparison of all nine research paper is as follow which is help to finalize the research work.

Sr. No.	Ref. No.	Food Item	Pesticides Analysed	Method
1	[27]	Rice	5 Pesticides	Equipment-Water alliance-TQD with Electrospray Ionization. LOQ-25 ppb Column C18, 5µm × 4.8 mm × 250 mm Flow-0.5 ml/min. Run Time-25 Min. Mobile Phase- Acetonitrile: Water (50:50 v/v) + 0.5 % formic acid
2	[28]	Rice whole grain and rice straw	Neonicotinoids	Equipment- LOQ- Column C18, 5µm × 4.8 mm × 250 mm Flow-0.6 ml/min.

				Run Time-45 Min. Mobile Phase
3	[29]	Fruits and vegetables	300 Pesticide residues	Equipment-Agilent LC-MS/MS LOQ:10 ppb Column: C18, 4µm × 3 mm × 100 mm Flow: 0.5 ml/min. Run Time: 30 Min. Injection Volume: 10 µl Mobile Phase: A-0.02 M ammonium acetate in Water. B-0.02 M ammonium acetate in Methanol
5	[30]	Cereal products	39 Pesticide residues	Equipment: SHIMADZU-LC-MS/MS LOQ: 10 ppb Column: C18, 5µm × 2 mm × 150 mm Flow: 0.5 ml/min. Run Time: 25 Min. Method Validation Protocol: SANTE/12982/2019. Standard solutions stored at – 20 °C
6	[31]	Cherries	8 Pesticide residues	Equipment-AB SCIEX 4000 QTRAP LOQ-10 ppb Column C18, 3µm × 2 mm × 50 mm Flow-0.3 ml/min. Run Time-30 Min. Mobile Phase- A-0.005 M ammonium acetate in Water. B-0.005 M ammonium acetate in Methanol

				0.1 % formic Acid each. Method Validation Protocol: SANTE/11813/2017. Standard solutions stored at – 20 °C
7	[32]	Oats	60 Pesticide residues	Equipment-Varian-LC-MS/MS LOQ-25 ppb Column C18, 3.5 µm × 3 mm × 50 mm Flow-0.5 ml/min. Run Time-25 Min. Mobile Phase-5 Mm Method Validation Protocol: SANTE/11813/2017. Standard solutions stored at – 20 °C
8	[33]	Cereals	70 Pesticide residues	Equipment-Water alliance-TQD with Electrospray Ionization. LOQ-25 ppb Column C18, 5µm × 4.8 mm × 250 mm Flow-0.5 ml/min. Run Time-25 Min. Mobile Phase- Water having 0.1 % Acetic acid and organic is methanol (30-Water: 70-Methanol).Method Validation Protocol: SANTE/11813/2017. Standard solutions stored at – 20 °C
9	[34]	Cereals	250 Pesticide residues	Equipment-AB SCIEX LC-MS/MS Column C18, 5µm × 4.8 mm × 100 mm Flow-0.5 ml/min. Run Time-24 Min. Mobile Phase- A - 0.005 M ammonium acetate in Water. B - 0.005 M ammonium acetate in

				Methanol

3. Food Contamination and method for Testing

Contamination and adulteration with respect to food products has become a major health issues now a days across globe. Food adulteration is the intentional approach to degrade the quality of food or adding inferior products to the food items while food contamination refers to the presence of external particle like microorganisms (bacteria, virus, germs, pathogens), chemical substances, physical contaminants like glass, wood, stone, metal, insects and plastics, etc. In some cases, few allergens are also found to be present in food items like in milk, eggs, peanuts, etc. Recently various situations associated to nutrition contamination and food safety have reported, which has raised the concern of health issues related to human being. In the analytical world, there is a lack of rapid, simple method of estimation of pesticide residue on crops [35-36]. Use of pesticide formulation to enhance the productivity by controlling different pest which damage the crops.

In India Food Safety Standard Authority of India play very vital function to regulate the contamination or adulteration via chemical, certain additives and presence of infectious microbes in food items. However, different type of chemical contamination is detected in food such as mycotoxins (Aflatoxins, Patulin, Ochratoxin A, Don etc.), Heavy metal, Antibiotics, Additives, Poly chlorinated biphenyl (PCBs), Polycyclic Aromatics Hydrocarbon (PAHs).United Nations sustainable development goal number three, which seeks a substantial reduction of illness from chemical contamination by 2030 [37].

To identify pesticide residue in cereals like rice, need to have good analytical separation technique like LC-MS/MS which is widely used instrument for detection of pesticide residue in rice. There number pesticide formulation register under Central Insecticides Board & Registration Committee, Ministry of Agricultural and farmer welfare, Department of Agriculture and Farmer Welfare, Directorate and plant protection and quarantine and storage, Government of India. For such multi residue pesticide detection LC-MS/MS instrument is needed.

In my research, I will work on to develop short and robust instrumental method. In the above some reviewed researched paper QuEChERS (Quick, Easy, Cheap, Effective, Rugged and safe) method had been implemented for sample preparation and having number of instrumental method but they are not commercially feasible to implement, hence I would like to develop more commercial method which has short run time, with good recovery, repeatability, and reproducibility and robust. After developing the method, I will analyse the rice sample which are export to EU country from India and compare the result with Indian guideline and international guideline.

4. Objectives of Research:

Following objectives are set for research work:

4.1 To develop a more efficient and sensitive technique on LC-MS-MS instrument for detecting pesticide residues in the rice grain.

4.2 To validate the proposed technique in alignment with EU/11312/2021.

4.3 To identify and quantify the pesticide residues in various rice matrices offering a complete contamination profile of the pesticides contamination in rice matrix.

4.4 To assess the pesticides contamination levels as per the Food Safety and Standards Authority of India, Ministry of Health and Family Welfare, GoI, and international regulations such as European Union Regulations and Codex Alimentarius Commission for residues of pesticides to ensure national and global adherence, respectively.

5. Methodology:

By LC-MS/MS instrument

Different steps includes in methodology which are as follows:

5.1 Development of LC-MS/MS Technique:

5.1.1 Selection of LC-MS/MS Instrument based on sensitivity, sufficiency and suitability for pesticide residues analysis.

5.1.2 Optimization of chromatographic condition, including column selection, mobile phase selection and gradient elution program to achieve optimal separation and detection of pesticide residue.

5.1.3 Development of MS-MS parameters, including ionization mode, collision energy and multiple reaction monitoring (MRM) transition, for sensitive and selective detection of target pesticides.

5.2 Validation of Proposed Technique:

5.2.1 Conduction the method validation studies following the guideline in EU/11312/2021 for pesticide residues analysis.

5.2.2 Evaluation of validation parameter such as linearity, LOQ (Limit of quantification, matrix effect, accuracy, precision, robustness Ion ratio and retention time of developed LC-MS/MS method.

5.2.3 Comparison of the results obtained using the proposed technique with established reference methods to ensure its accuracy and suitability of pesticide residue analysis in rice sample.

5.3 Determination and Quantification of Pesticide residues:

5.3.1 Extraction of pesticide residues from rice matrix using the suitable method.

5.3.2 Analysis of extracted samples using the validated LC-MS/MS method to identify and quantify pesticide residues present in rice matrices.

5.3.3 Generation of a comprehensive contamination profile of pesticides including the identification of individual pesticide residues and assessment of their occurrence levels across different rice samples.

5.4 Comparison of Pesticide Residue Level:

5.4.1 Comparison of pesticide residue detected in rice sample with the tolerance set by regulating authorities.

5.4.2 Adherence to international regulations established by organizations such as the European Union Regulations and the Codex Alimentarias Commission for Residues of Pesticides to a ensure compliance with global standards for food safety and quality.

5.4.3 Statistical analysis of the data to identify any significant variation in pesticide occurrence and levels among different rice matrices and geographical regions.

5.5 Sampling, Sampling frequency and Location:

5.5.1 Samples will be collected randomly from different vendor of local market. Sampling location will be New Delhi, Haryana, Uttar Pradesh and Punjab states.

5.5.2 Sampling frequency will be every month with 12 different vendor from each location for six months.

5.5.3 Samples will be collected randomly from different vendor of local market.

5.5.4 Total number of samples analyze will be around 288.

5.6 Instrumentation:

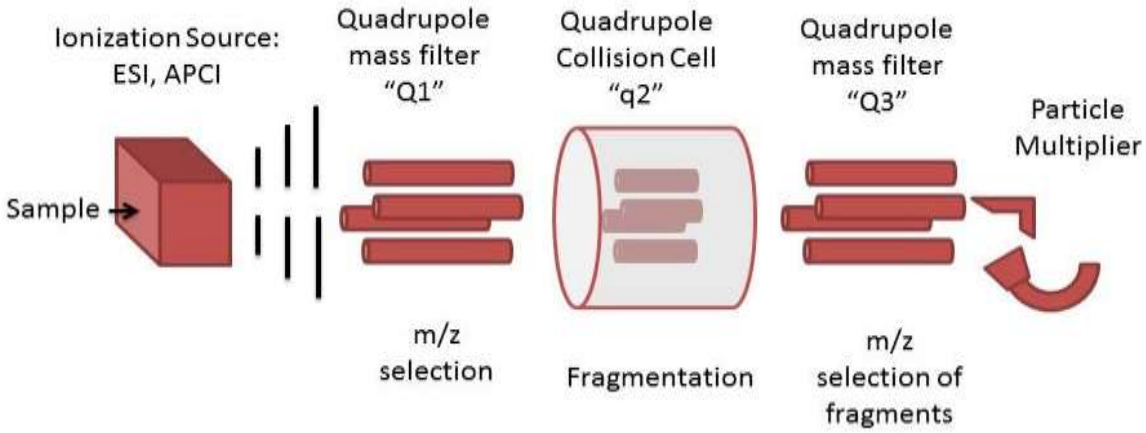

Fig.1 Schematic diagram of LC-MS/MS

Mass spectrometry is pertaining to the generation of gaseous ions from analyte molecules, the subsequent separation of these ions according to their mass-to charge ratio (m/z), and detection of these ions. The resulting mass spectrum is a plot of the abundance of the ions produced as a function of the m/z ratio. It consists of five parts:

1. Sample injection.
2. Ionization.
3. Mass Analyzer.
4. Ion Detector
5. Data processing.

5.6.1 Sample introduction and ionization:

Sample entered in to the MS from column and get evaporated and ionized with the help of heated nitrogen and different voltages.

Classification of ionization techniques for mass Spectrometry

S. No.	Sample state	Ionization Technique	Example	Full form
1	Gas or Vapor	Electron ionization	EI	Electron ionization
2	Gas or Vapor	Chemical ionization	CI	Chemical ionization

			DCI	Desorption Chemical ionization
3	Liquid or Solid	Desorption	FAB	Fast –atom bombardment
			SIMS	Secondary ion MS
			FD	Field desorption
			PD	Plasma desorption
			MALDI	Matrix –assisted laser desorption/ionization
4	Solution	Other	TSI	Thermospray ionization
			ESI	Electrospray ionization
			APCI	Atmospheric Pressure Chemical Ionization

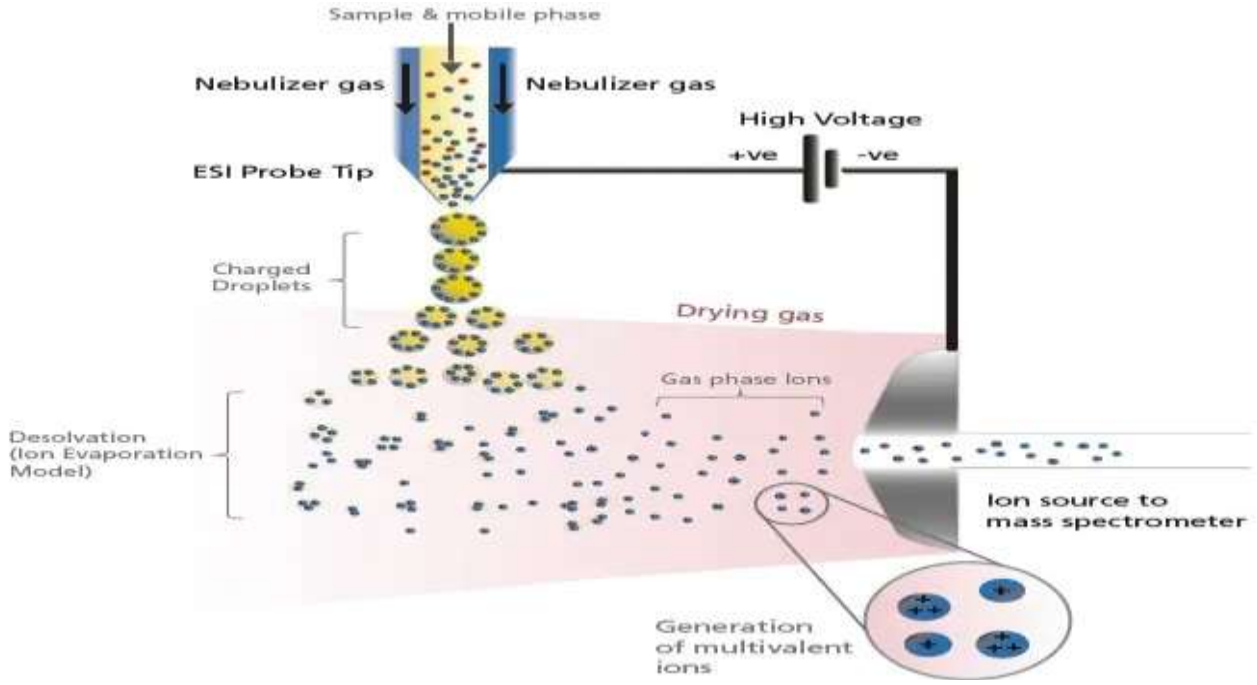

Fig.2 Schematic diagram of Sample introduction and ionization

5.6.2 Mass Analyzer:

Four different principle of mass analysis are applied in MS instruments:
1. A combination of magnetic and electric sector
2. Quadrupole filters
3. Time of Flight Measurement
4. Ion –cyclotron system.

5.6.2.1 Quadrupole filters:

The ion source generates the ion beam which is introduced in ion chamber of quadrupole mass filter for further disintegration into their ions. These ions generated in the quadrapole starts to oscillate in perpendicular plane and follow the trajectories where it gets separated on the basis of stable m/z; once these ions migrate towards the detector, their m/z values can be interpreted.

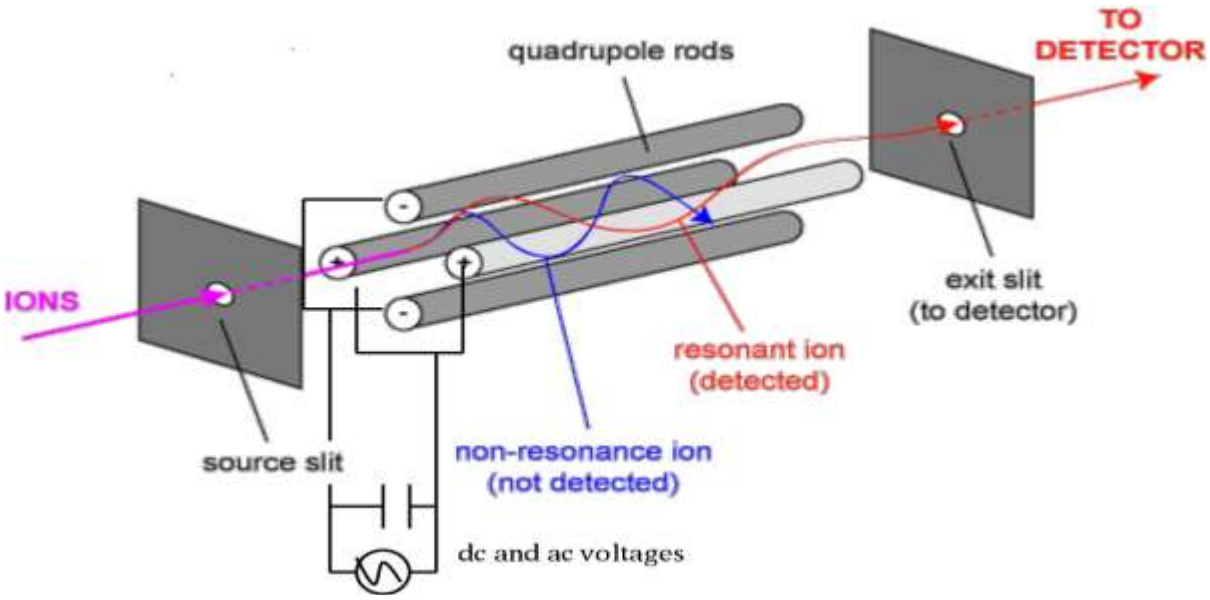

Fig.3 Internal layout of quadrupole filters

5.6.3 Ion Detector:

The detection of ions done by an electron multiplier which is based on the emission of secondary electrons consequently from the collision of energetic particles at a suitable surface. The secondary electrons can be multiplied by consecutively striking subsequent surface. The electron multiplier may be either of the discrete dynode or of the continuous dynodes. A discrete dynodes multiplier comprised of 12-20 beryllium –copper dynodes, electronically connected through a resistive network.

5.6.3.1 Discrete dynode:

Positive point of using this includes a response time in the picoseconds, a high sensitivity, and an electron gain of about 10^6 electrons.

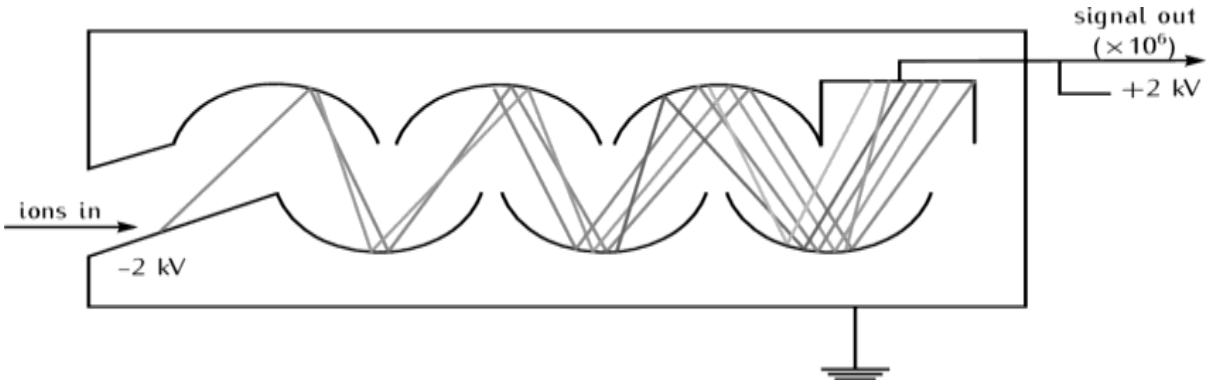

Fig.4 Discrete dynode

5.6.3.2 Continuous dynode:

A continuous-dynode electron multiplier (CEM) is a type of electron multiplier that has a single electrode in the shape of a horn. The horn is made from lead-silicate glass coated with a semi-conductive substance, such as lead oxide. When ions hit the open end of the horn, they create a cascade of electrons that are multiplied as they interact with the horn's wall. The electrons are then focused onto a preamplifier. CEMs operate on the same principle as discrete-dynode electron multipliers, but instead of having discrete metal dynodes, they have a continuous inner surface that emits electrons. The inner surface's film acts as a spatial resistance, so CEMs don't have discrete resistors. CEMs are known for their high amplification, fast time response, low noise, and high collection efficiency.

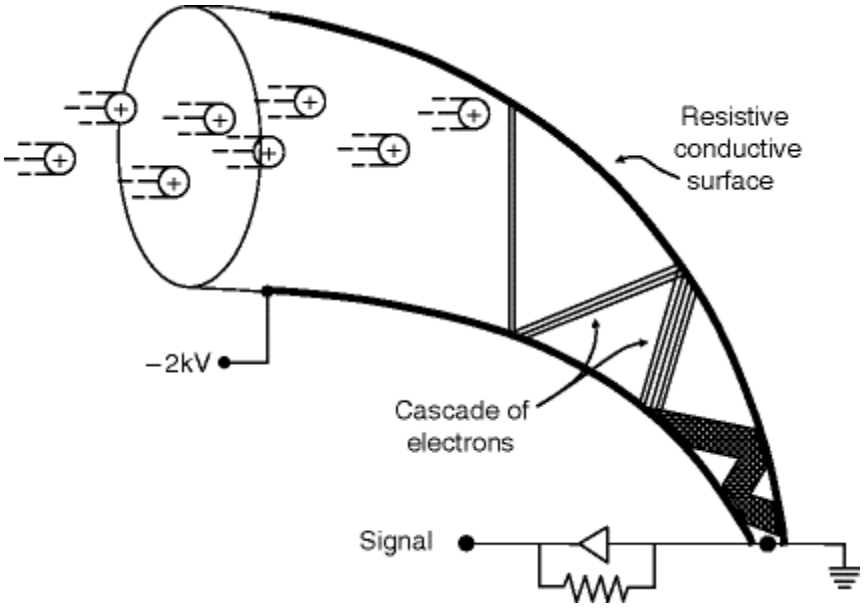

Fig.5 Continuous dynode

5.6.4 Data processing:

Computerized data handling is a prerequisite in mass spectrometry. During the acquisition, the instrument is sweeping the AC and DC potentials at a fixed ratio in order to transmit ions in the range of m/z 50-500.Such scanning is made every second or few seconds. The three-dimensional data-array produced during acquisitions can be handled in various ways. The most important types of output are:

5.6.4.1 Chromatogram for the total ion:

Following chromatogram is for total ion of respective compound which plotted counts vs acquisition time.

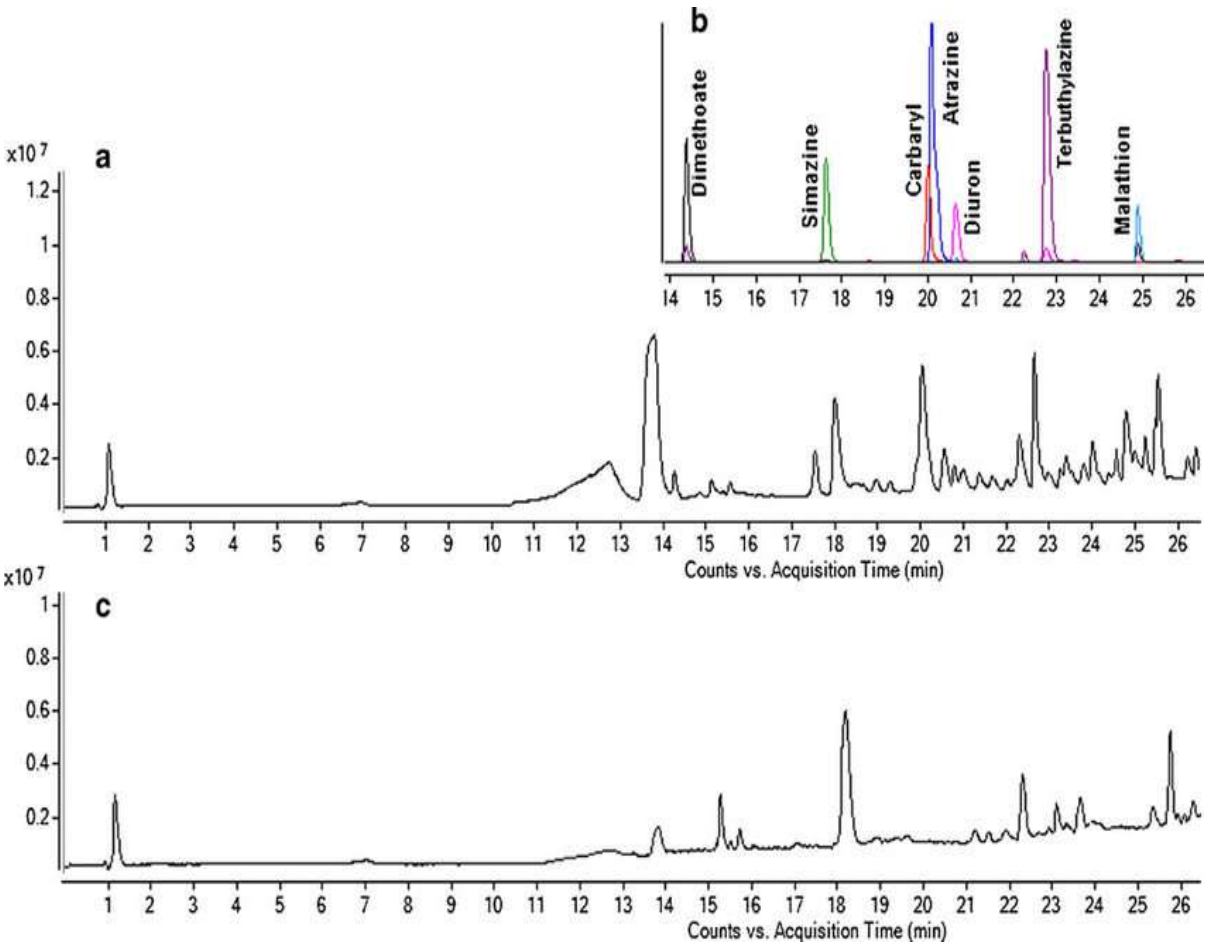

Fig.6 The total-ion chromatogram

5.6.4.2 The Mass Chromatogram:

The ion intensity for an ions with particular m/z is plotted as a function of time or scan number.

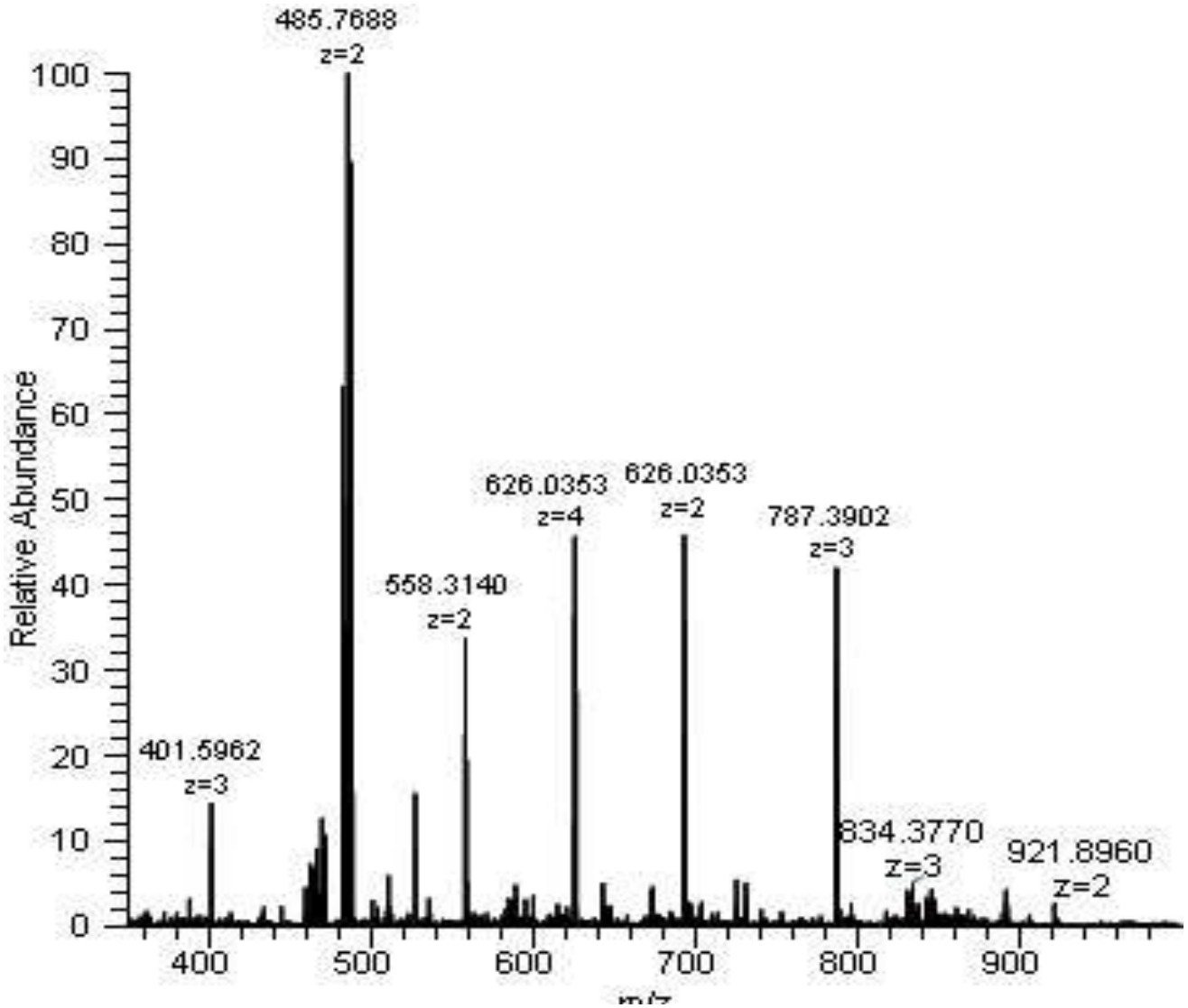

Fig.6 The Mass Chromatogram

5.6.4.3 The mass spectrum:

Following is the mass spectra for Imidacloprid that showing fragmented mas with parent mass.

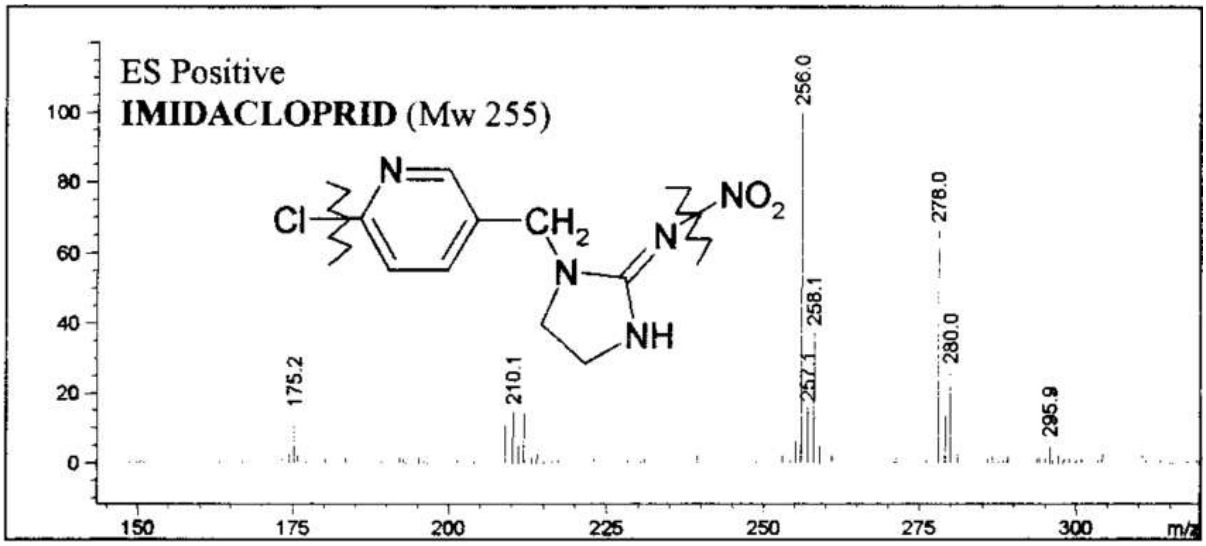

Fig.6 The mass spectrum-Imidacloprid with Mw is 255.

6. Expected Outcome of the Research:

The outcomes of the study are expected to be beneficial. The validated of a single method capable of analyzing numerous pesticide residues in food matrices will be a significant achievement. The existing written work review has revealed a gap in methodologies capable of detecting pesticide residues at trace levels, which is both commercially viable and time-efficient. Current methods often entail lengthy analysis times, necessitating the use of multiple columns, mobile phases, and equipment, thereby presenting limitations.

The proposed research aims to address these challenges by developing a more efficient method validated according to international guidelines. This validation will encompass parameters such as specificity, matrix effect, limit of quantification (LOQ), recovery, repeatability, reproducibility, robustness, ion ratio, and retention time. By adhering to these stringent criteria, the developed method is poised to become widely accepted for the detection of pesticide residues in food, particularly rice.

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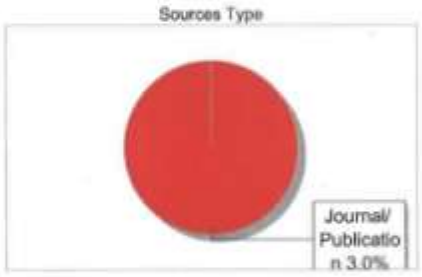

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STUDY OF THE PESTICIDE RESIDUES IN RICE MATRIX BY LIQUID CHROMATOGRAPHY COUPLED WITH MASS SPECTROMETER

SHORT SYNOPSIS

by

Deepak Ashok Temak
MRUCHDN012K22013

Under the supervision of

Dr. Meena Kapahi
Dr. Roopa Rani

MANAV RACHNA UNIVERSITY

Year 2025

Abstract

To determine pesticide residue in rice matrices at low concentration, there is a need for suitable analytical method. In the current study, the dispersive solid-liquid extraction method has been employed to monitor different types of pesticides by liquid chromatography with tandem mass spectrometry (LC-MS/MS) electrospray ionization (ESI) technique with a minimum run time. The QuEChERS method for pesticide residues extraction and determination in cereals is used. More than 50 different pesticides are monitored for their compliance with it tolerance set by different regulatory bodies. The method validation protocol is utilized as per SANTE - 11312/2021 guidelines by evaluating different parameters such as linearity, specificity, LOD, LOQ, recovery, repeatability, reproducibility, ion ratio and robustness.

Keywords: Pesticide Residue, Dispersive Solid-Liquid Extraction, LC-MS/MS, EU Guideline, SANTE 11312 guideline, Maximum Residual Limit, QuEChERS

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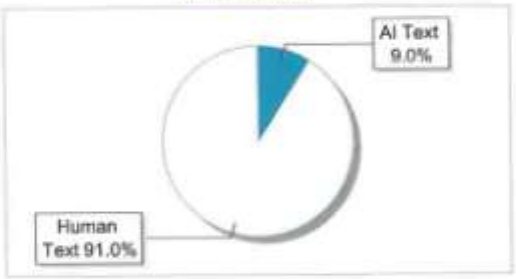

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